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Year: 2017

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## Effect of immediate dentine sealing on the fracture strength of lithium disilicate and multiphase resin composite inlay restorations

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**Abstract:** PURPOSE Limited information is available on the effect of Immediate Dentin Sealing (IDS) on the fracture strength of indirect partial posterior restorations. This study evaluated the effect of IDS on the fracture strength and failure types of two indirect restorative materials. MATERIALS AND METHODS Standard MOD inlay preparations were made on sound molars (N=40, n=10 per group) and randomly divided into four groups to receive the inlay materials with and without the application of IDS: Group L-IDS-: Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> (Lithium disilicate, IPS e.max) without IDS; Group L-IDS+: Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> with IDS; Group MR-IDS-: Multiphase resin composite (MR, Lava Ultimate) without IDS; MR-IDS+: MR with IDS. Inlays made of L were etched with 5% hydrofluoric acid, and MR inlays were silica coated. After silanization, they were cemented using adhesive resin cement (Variolink Esthetic DC). The specimens were thermo-mechanically aged (1.2×10<sup>6</sup> cycles, 1.7Hz, 8000 cycles, 5-55°C) and then subjected to load to failure (1 mm/min). Failure types and locations of debondings were classified. Data were statistically analyzed using ANOVA, Mann Whitney U-test and Chi-square tests ( $\alpha=0.05$ ). Two-parameter Weibull distribution values including the Weibull modulus, scale (m) and shape (0), values were calculated. RESULTS After aging conditions, no apparent changes were observed in marginal integrity but occlusal wear facets were more common with MR than with L ( $p<0.001$ ). Material type and the application of IDS significantly affected the results ( $p=0.013$ ). While group L-IDS- showed the lowest mean fracture strength (1358±506N) among all groups ( $p<0.05$ ), application of IDS significantly increased the results significantly (L-IDS+: 2035±403N) ( $p=0.006$ ). MR groups with and without IDS, did not show significant difference (MR-IDS-: 1861±423, MR-IDS+: 1702±596 N) ( $p=0.498$ ). When materials without IDS are compared, L showed significantly lower results than that of MR ( $p=0.035$ ). With the application of IDS, no significant difference was noted between L and MR materials ( $p=0.160$ ). Weibull distribution presented the highest shape (0) for L-IDS+ (5.66) compared to those of other groups (3.01-4.76). Neither the material type ( $p=0.830$ ), nor the application of IDS ( $p=0.54$ ) affected the severity of the failure types. In 95% of the cases, the IDS layer left adhered on the tooth surface after fracture tests. In groups where no IDS was used, resin cement remained on the tooth surface in 44% of the cases ( $p=0.001$ ). No significant differences were observed between the materials with respect to cement remnants or IDS after fracture ( $p=0.880$ ). The incidence of repairable failure types (83%) was more common with L than with MR (75%) material ( $p>0.05$ ). CONCLUSION Immediate dentin sealing improves adhesion, and thereby the fracture strength of inlays made of lithium disilicate but not that multiphase resin composite.

DOI: <https://doi.org/10.1016/j.jmbbm.2017.04.002>

Posted at the Zurich Open Repository and Archive, University of Zurich

ZORA URL: <https://doi.org/10.5167/uzh-145984>

Journal Article

Accepted Version



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Originally published at:

van den Breemer, Carline R G; Özcan, Mutlu; Cune, Marco S; van der Giezen, Rianne; Kerdijk, Wouter; Gresnigt, Marco M M (2017). Effect of immediate dentine sealing on the fracture strength of lithium disilicate and multi-phase resin composite inlay restorations. *Journal of the Mechanical Behavior of Biomedical Materials*, 72:102-109. DOI: <https://doi.org/10.1016/j.jmbbm.2017.04.002>

**Effect of immediate dentine sealing on the fracture strength of lithium disilicate  
and  
multiphase resin composite inlay restorations**

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**Short title:** *Effect of immediate dentin sealing on durability of inlays*

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## ABSTRACT

**Objectives.** Limited information is available on the effect of Immediate Dentin Sealing (IDS) on the fracture strength of indirect partial posterior restorations. This study evaluated the effect of IDS on the fracture strength and failure types of two indirect restorative materials.

**Methods.** Standard inlay preparations were made on sound molars (N=40, n=10 per group) and randomly divided into four groups to receive the inlay materials with and without the application of IDS: Group L-IDS<sup>-</sup>: Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> (Lithium disilicate, IPS e.max) without IDS; Group L-IDS<sup>+</sup>: Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> with IDS; Group MR-IDS<sup>-</sup>: Multiphase resin composite (MRC, Lava Ultimate) without IDS; MR-IDS<sup>+</sup>: MR with IDS. Inlays made of L were etched with 5% hydrofluoric acid, and MR silica coated. After silanization, they were cemented using adhesive resin cement (Variolink Esthetic DC). The specimens were thermo-mechanically aged (1.2x10<sup>6</sup> cycles, 1.7 Hz, 8000 cycles, 5-55°C) and then subjected to load to failure (1 mm/min). Failure types and locations of debondings were classified. Data were statistically analyzed using ANOVA, Mann Whitney U-test and Chi-square tests ( $\alpha=0.05$ ). Two-parameter Weibull distribution values including the Weibull modulus, scale ( $m$ ) and shape ( $0$ ), values were calculated.

**Results.** After aging conditions, no apparent changes were observed in marginal integrity but occlusal wear facets were more common with MR than with L ( $p<0.001$ ). Material type and the application of IDS significantly affected the results ( $p=0.013$ ). While group L-IDS<sup>-</sup> showed the lowest mean fracture strength (1358±506 N) among all groups ( $p<0.05$ ), application of IDS significantly increased the results significantly (L-IDS<sup>+</sup>: 2035±403 N) ( $p=0.006$ ). MR groups with and without IDS, did not show significant difference (MR-IDS<sup>-</sup>: 1861±423, MR-IDS<sup>+</sup>: 1702±596 N) ( $p=0.498$ ). When materials without IDS are compared, L-IDS<sup>-</sup> showed significantly lower results than that of MR-IDS<sup>-</sup> ( $p=0.035$ ). With the application of IDS, no significant difference was noted between

L and MR materials ( $p=0.160$ ). Weibull distribution presented the highest shape ( $\alpha$ ) for L-IDS<sup>+</sup> (5.66) compared to those of other groups (3.01-4.76). Neither the material type ( $p=0.830$ ), nor the application of IDS ( $p=0.54$ ) affected the severity of the failure types. In 95% of the cases, the IDS layer was left adhered on the tooth surface after fracture tests. In groups where no IDS were used, resin cement remained on the tooth surface in 44% of the cases ( $p=0.001$ ). No significant differences were observed between the materials with respect to cement remnants or IDS after fracture ( $p=0.880$ ). The incidence of repairable failure types (83%) was more common with L than with MR (75%) material ( $p>0.05$ ).

**Significance.** Immediate dentin sealing improves adhesion, and thereby the fracture strength of inlays made of  $\text{Li}_2\text{Si}_2\text{O}_5$  but not that multiphase resin composite.

**Keywords:** Cement; Ceramic; Cyclic loading; Fracture strength; Immediate dentin sealing; Indirect composite; Inlay; Lithium disilicate; Multiphase resin composite.

## 1. Introduction

Minimally invasive dentistry strives for preservation of enamel as much as possible since removing large amount of tooth structure has an adverse effect on the pulp and may lead to vitality loss [1,2]. Current restorative concepts are based on bio-emulation philosophy that is to restore teeth mimicking both the biomechanical and structural properties of a natural tooth [3-5]. By taking biology, mechanics, function and aesthetics into account, a harmonious and natural restorative result could be achieved [6]. A restoration following these principles can either be made from a direct or an indirect restorative material, where the former is chosen when restoration of morphology and function is difficult to restore [4,7,8]. A restoration made of an indirect restorative material that suits a minimally invasive preparation is a so-called partial restoration. In the application of indirect partial restorations several components are relevant, namely the material, adhesive cementation to dentin/ enamel, and the bonding procedures.

Indirect partial restorations could be made of various materials. Gold onlays are reliable restorative options with success rate of 92% over nine years [6,9-11]. Studies on ceramic indirect restorations show survival rate between 90 and 100% after five years [6,9,12-14], and a success rate between 89 and 91% after ten years [7,14]. On the other hand, multiphase resin composite restorations present three-year survival rate of 100% [15], while others reported a two-year survival rate of 90% [15], with an average annual failure rate of 0 - 11.8% [12].

Limited information is available on the clinical survival of indirect resin composites [14-18]. Likewise, little is known on the multiphase resin composite (MR, Lava Ultimate, 3M ESPE) but it is claimed that restorations made of this material have comparable fatigue resistance with those made of ceramics [19]. The most common cause of failure of inlays made of either ceramic or resin composite is fracture [11,12,14,20-23]. Such fractures are primarily within the restorative material, followed by fractures in the tooth

[7,16,20,21]. In fact, adhesive cementation provides chemical and micro-mechanical attachment of the restoration to the tooth and re-establishes the integrity of the tooth and circumvents microleakage [2,24,25]. In that respect, sealing the dentin immediately after tooth preparation using a dentin bonding agent to the freshly cut dentin, the so called 'immediate dentin sealing (IDS)', was advocated in early 1990s [26]. Several studies have shown that application of IDS after tooth preparation ensure improved bond strength of resin based materials [5,27,28] and ceramic restorations to dentin [5,28-34]. However, it is ambiguous whether the application of IDS would have a similar positive effect on the fracture strength of inlays.

The objective of this study therefore was to compare the fracture strength of lithium disilicate ( $\text{Li}_2\text{Si}_2\text{O}_5$ ) and multiphase resin composite material with and without the application of IDS. The hypothesis tested was that the presence of IDS would positively contribute to the fracture strength of the glass ceramic and the indirect resin composite material compared to conventional adhesive cementation.

## **2. Material and methods**

### ***2.1 Specimen preparation***

The brands, types, manufacturers, chemical compositions and batch numbers of the materials used for the experiments are listed in Table 1. Schematic description of the experimental design is presented in Fig. 1.

Sound human molars (N=40) of similar size, free of restorations, fractures, caries and root canal treatment were selected from a pool of recently extracted teeth (<6 months). All teeth were screened on the presence of cracks through blue light illumination and those with cracks were eliminated. The selected teeth were placed in polyvinylchloride (PVC) tubes (height: 10 mm; diameter: 15 mm) and filled with polymethylmethacrylate (Probase Cold, Ivoclar Vivadent, Schaan, Liechtenstein) up to 1 mm below the cement-enamel junction (CEJ). After photographs were made from each specimen, they were

scanned using an intraoral scanner (Lava 3M ST scanner, 3M ESPE, St. Paul, USA). The scanned images served for the definitive form of the restorations after preparation. Specimens were stored in distilled water at 37°C during the experiments.

Teeth were randomly divided into four groups to receive the inlay materials with and without the application of IDS: Group L-IDS<sup>-</sup>: Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> (Lithium disilicate, IPS e.max, Ivoclar Vivadent, Schaan, Liechtenstein) without IDS; Group L-IDS<sup>+</sup>: Li<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> with IDS; Group MR-IDS<sup>-</sup>: Multiphase resin composite (MRC, Lava Ultimate, 3M ESPE, St. Paul, USA) without IDS; MR-IDS<sup>+</sup>: MR with IDS.

Standard preparations were made in each tooth (bucco-lingual width: 5 mm, depth: 2 mm from the fissure, approximal outline: 1 mm above the CEJ) using different burs (no. 6856,8856, TPS2-8, TPS2-9, Komet Dental, Lemgo, Germany). The width of the preparation determined the **diameter** of the remaining walls, depending on the size of the tooth. The axial walls were prepared with divergence of <6° to eliminate undercuts. The dimensions of the preparations were checked using an electronic caliper and adjusted after the preparation where needed.

## **2.2 Immediate dentin sealing**

In groups L-IDS<sup>+</sup> and MR-IDS<sup>+</sup>, IDS was applied immediately after tooth preparation. A self-etching primer (Clearfil SE Bond, Kuraray Co., Tokyo, Japan) was actively applied to the dentin surface for 20 s, air-dried gently with oil-free air, until dry and glossy appearance of the dentin was maintained. Hereafter, an adhesive resin (Clearfil SE Bond, Kuraray Co.) was applied with microbrush on the dentin only and photo-polymerized for 10 s using an LED polymerization device (Bluephase 20i, Ivoclar Vivadent) from a distance of 2 mm. Then, flowable resin composite (Tetric Evoflow, Ivoclar Vivadent) was applied on the dentin surface in order to increase the thickness and protect the IDS layer and photo-polymerized for 40 s. The output of the polymerization device was >1000mW/cm<sup>2</sup> throughout the experiment verified by a radiometer (Bluephasemeter, Ivoclar Vivadent). After application of glycerine gel



(Johnson & Johnson, Sezanne, France), the surface was again photo-polymerized for 40 s. Excess adhesive resin on enamel was removed using a fine grid diamond bur (no. 862EF, Komet Dental, Lemgo, Germany) and rubbers (no. 9619, Komet Dental) under an operatory microscope (x10 Opmpico, Zeiss, Oberkochen, Germany). Digital photos were made from 5 sides and then the teeth were scanned again using an intraoral scanner (Lava 3M ST Scanner, 3M ESPE) after which the STL files were sent to the dental laboratory.

### ***2.3 Temporary and permanent restorations***

In groups L-IDS<sup>+</sup> and MR-IDS<sup>+</sup>, glycerine gel (Johnson & Johnson) was applied on the IDS layer before placing the provisional restorations in order to prevent adhesion between IDS and the provisional material (Protemp 4, 3M ESPE) [35]. Provisional restorations were adjusted using polishing discs (Sof-Lex Contouring and Polishing Disks, 3M ESPE) and luted with temporary cement (Durelon, 3M ESPE). Specimens were stored in distilled water at 37°C for 3 weeks.

One dental technician fabricated lithium disilicate inlays according to the instructions of the manufacturer. Ceramic restorations were milled in wax and then pressed and glazed in a ceramic oven (Programat EP5000, Ivoclar Vivadent) while multiphase resin composite restorations were milled in a 5-axis milling machine (Lava 3M CNC 500, 3M ESPE) and glazed.

### ***2.4 Adhesive cementation***

After removing the provisional restorations, each tooth was cleaned with pumice and the fit of the ceramic restorations was controlled using an optical microscope (x10, OpmiPico, Zeiss). A dual-polymerized resin composite cement (Variolink Esthetic DC, Ivoclar Vivadent) was used for cementation of the ceramic restorations. A two-step bonding procedure (Adhese Universal, Ivoclar Vivadent) with separate conditioning of the IDS layer was employed to ensure adhesion.

Cementation surfaces of the ceramic inlays were conditioned using 5% hydrofluoric acid (IPS Ceramic etching gel, Ivoclar Vivadent) for 20 s, rinsed with water and a neutralizing powder (IPS Ceramic neutralizing powder, Ivoclar Vivadent). The restorations were ultrasonically cleaned (Emag, Valkenswaard, the Netherlands) in distilled water for 5 minutes. Hereafter the restorations were dried, silanized (Monobond Plus, Ivoclar Vivadent) and hot air-dried for 60 s. Adhesive resin was applied (Adhese Universal, Ivoclar Vivadent) to the ceramic surfaces, air-thinned but not photopolymerized.

Cementation surfaces of the resin composite restorations were tribochemically treated (CoJet Sand, 3M ESPE) for 10 s with nozzle angle of 45°, distance of 10 mm at 2 bar pressure using a chairside air-abrasion device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark). Silane coupling agent (ESPE-SIL, 3M ESPE) was applied on the adhesion surface, left to react for 5 minutes and hot air-dried for 2 minutes. Adhesive resin was then applied (Adhese Universal, Ivoclar Vivadent) with a microbrush on the composite surface.

In groups L-IDS<sup>-</sup> and MR-IDS<sup>-</sup>, teeth were etched with 37% H<sub>3</sub>PO<sub>4</sub> (enamel: 30 s, dentin: 10 s, Total-etch, Ivoclar Vivadent) and rinsed with copious water for 30 s. In groups L-IDS<sup>+</sup> and MR-IDS<sup>+</sup>, the IDS layer was tribochemically treated (CoJet, 3M, ESPE) using a chairside air-abrasion device (Dento-Prep<sup>TM</sup>, RØNVIG A/S, Daugaard, Denmark) for 4 s with nozzle angle of 45°, distance of 10 mm at 2 bar pressure. Enamel was etched with 37% H<sub>3</sub>PO<sub>4</sub> for 30 s, rinsed and air-dried. Silane (ESPE-Sil, 3M ESPE) was applied one coat on the silica-coated IDS surfaces left to react for 5 minutes. Subsequently, adhesive resin (Adhese Universal, Ivoclar Vivadent) was applied to the whole preparation using a microbrush for 20 s.

Inlay restorations were cemented using dual-polymerized resin composite cement (Variolink Esthetic DC, Ivoclar Vivadent) and excess cement was removed with microbrushes. Glycerine gel (Johnson & Johnson) was applied at the margins of the

restorations and photo-polymerized for 40 s from labial, lingual and incisal sides (Bluephase, Ivoclar Vivadent, light output:  $\geq 1000 \text{ mW/cm}^2$ ). Margins were polished using sofex discs (Sof-lex Contouring and polishing disks, 3M ESPE) and rubber burs (no. 9619, Greenies, Komet Dental).

### ***2.5 Aging and fracture test***

All specimens were artificially aged in a chewing simulator (SD Mechatronik CS-4.8 Chewing Simulator, Feldkirchen-Westerham, Germany) using a ceramic antagonist sphere (50 N) on the occlusal plane for  $1.2 \times 10^6$  cycles, 1.7 Hz) and hydrolytically aged ( $\times 8000$  cycles between 5-55°C) in distilled water. Changes in marginal gap and occlusal wear were evaluated after thermo-mechanical loading under optical microscope ( $\times 40$ , Leica Wild Heerbrugg, M3Z Schott Zeiss KL200).

The specimens were then mounted in the jig of the Universal Testing Machine (810 Material Test System, MTS, Eden Prairie, USA) and loaded with 8 mm steel ball perpendicular to the occlusal surface at a crosshead speed of 1 mm/min. The maximum force to produce fracture was recorded.

### ***2.6 Failure analysis***

Failure sites were initially observed using an optical microscope (Leica Wild Heerbrugg, M3Z Schott Zeiss KL200) at  $\times 40$  magnification and classified as an ordinal variable with increasing severity as follows: Score 1: Fracture of the inlay; Score 2: Fracture of the inlay and enamel; Score 3: Fracture of the inlay, enamel and dentin, Score 4: Root fracture. The presence of the cement or IDS was also noted on the tooth after fracture. Failure types were further classified depending on their reparability where root fractures and deep subgingival fractures were scored as not repairable. Additionally, representative specimens from each group were sputter-coated with a 3 nm thick layer of gold (80%) / palladium (20%) (90 s, 45mA; Balzers SCD 030, Balzers, Liechtenstein) and analyzed using cold field emission Scanning Electron Microscope (SEM) (LEO 440, Electron Microscopy Ltd, Cambridge, United Kingdom).

## 2.7 Statistical analysis

Data were analyzed using a statistical software package (SPSS 22, PASW statistics 18.0.3, Quarry Bay, Hong Kong, China). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were normally distributed, 2-way analysis of variance (ANOVA) and Tukey's tests were applied to analyze possible differences between the groups for the parameters of material type and the effect of IDS on fracture strength results. Mann-Whitney U and Chi-Square tests were used to investigate differences in failure types between the experimental groups. Maximum likelihood estimation without a correction factor was used for 2-parameter Weibull distribution, including the Weibull modulus, scale ( $m$ ) and shape ( $\sigma$ ), to interpret predictability and reliability of adhesion (Minitab Software V.16, State College, PA, USA).  $P < 0.05$  was considered to be statistically significant in all tests.

## 3. Results

After aging conditions, no apparent changes were observed in marginal integrity but occlusal wear facets were more common with MR than with L (Figs. 2a-b) ( $\chi^2(1) = 18.027$ ,  $p < 0.001$ ).

Mean fracture strength results showed significant difference between the groups ( $p < 0.05$ ). Material type and the application of IDS significantly affected the results (ANOVA;  $F(1,34) = 6.94$ ,  $p = 0.013$ ).

While group L-IDS<sup>-</sup> showed the lowest mean fracture strength ( $1358 \pm 506$  N) among all groups ( $p < 0.05$ ), application of IDS significantly increased the results significantly (L-IDS<sup>+</sup>:  $2035 \pm 403$  N) ( $t(16) = 3.164$ ;  $p = 0.006$ ). MR groups with and without IDS, did not show significant difference (MR-IDS<sup>-</sup>:  $1861 \pm 423$ , MR-IDS<sup>+</sup>:  $1702 \pm 596$  N) ( $t(18) = 0.691$ ,  $p = 0.498$ ) (Table 2). When materials without IDS are compared, L-IDS<sup>-</sup> showed significantly lower results than that of MR-IDS<sup>-</sup> ( $t(16) = 2.30$ ;  $p = 0.035$ ). With the

application of IDS, no significant difference was noted between L and MR materials ( $t(18)=1.47$ ;  $p=0.160$ ).

Weibull distribution presented the highest shape ( $\sigma$ ) for L-IDS<sup>+</sup> (5.66) compared to those of other groups (3.01-4.76) (Fig. 1).

Neither the material type (Mann-Whitney test;  $U=173$ ;  $p=0.830$ ), nor the application of IDS (Mann-Whitney test;  $U=160$ ;  $p=0.54$ ) affected the severity of the failure types (Table 3). In 95% of the cases, the IDS layer was left adhered on the tooth surface after fracture tests. In groups where no IDS were used, resin cement remained on the tooth surface in 44% of the cases ( $\chi^2(1)=11.77$ ,  $p=0.001$ ) (Figs. 3a-b). No significant differences were observed between the materials with respect to cement remnants or IDS after fracture ( $\chi^2(1)=0.023$ ;  $p=0.880$ ). The incidence of repairable failure types (83%) was more common with L than with MR (75%) material ( $p>0.05$ ).

#### **4. Discussion**

This study evaluated whether the application of immediate dentin sealing (IDS) could improve fracture strength of lithium disilicate and multiphase resin composite inlays in molar teeth after aging. The application of IDS was previously not investigated in posterior teeth restored with ceramic or indirect resin composites. Based on the results of the present study, since IDS significantly increased the fracture strength of ceramic inlays but not the composite ones, the hypothesis could be partially accepted.

Clinical studies on partial ceramic posterior restorations without the application of IDS show survival probability of 80 to 95% over a period of 10 years [14,36]. Reported failures were due to fracture of the ceramic material and reduction in margin quality [37]. Bulk fracture of ceramic materials in general is still a major reason for failure due to inherent fragility of the ceramics [14]. Hence, it is important to improve the fracture strength of the ceramic materials especially in the posterior teeth.

In a recent in vitro study, IDS application significantly increased the fracture strength of laminate veneers made of lithium disilicate bonded to large dentin substrates [38]. In this study, not the resin composite but the ceramic inlays benefitted from IDS application. IDS application in combination with flowable resin composite, could decrease the space available for the indirect restoration that may eventually also decrease the cohesive strength of the restorative material. In the multiphase resin composite group, most failures were restoration fractures. Manufacturer recommendations of this material state that 1.5 mm or more space should be available at the isthmus height. In this study, a depth of 2 mm from the fissure was established but due to the application of IDS with the flowable resin composite, the depth could have been decreased. The strength of multiphase resin composite could increase with increased isthmus dimensions that need further investigations.

Average bite forces range between 20 to 1000 N but during normal function, forces do not exceed 270 N [40]. Only some patients with signs of bruxism express higher masticatory forces [39]. With an average load to failure value of 1835 N almost all the restorations fulfilled the maximum expected chewing forces of 1000 N. Mean fracture strength results of this study (1300 - 2000 N) are not consistent with previous studies where 1600 to 2600 N were reported [24,28,40]. However, it has to be noted that in those studies no aging procedures were performed. The current study employed thermo-mechanical cyclic loading (50 N,  $1.2 \times 10^6$  cycles, 1.7 Hz, 5-55°C) that was postulated to represent five years of clinical function [41]. During such aging process, different levels of degradation processes could be expected for the ceramic and resin composite materials. Interfaces between the resin composite matrix and the silica coated inorganic fillers are more prone to hydrolytic degradation mainly at the adhesive interface [42]. The materials used in this study did not show significant difference in fracture strength in conditions where no IDS was applied. Studies on fracture strength

using similar materials presented comparable values (1250 to 1580 N) [24], or higher values (1614 to 2522 N) being more in favour of ceramic materials [43].

In this study, multiphase resin composite inlays showed significantly more visible occlusal wear than the lithium disilicate ones. Such wear facets may initiate crack formation already during cyclic loading. Typically, the antagonist material used in such aging procedures is made of enamel or ceramic [42,45]. In this study, ceramic was used as an antagonist sphere. When antagonist materials are compared, ceramic ones cause more wear (130-265  $\mu\text{m}$ ) than enamel (120-199  $\mu\text{m}$ ), especially when the tested material is composite [41,44]. The results may change when antagonist material is enamel or metal. Yet, the choice of ceramic may represent a worse-case scenario.

In the failure analysis it was noted that In the majority of the specimens IDS layer was still intact on the dentin surfaces after the fracture test. This implies that adhesion to dentin was in fact more stable than the adhesion of the resin composite to the intaglio surfaces of the inlays. When IDS was not used, the cement remained on the tooth in 44% of the cases. Thus, employing an IDS layer, the weakest link remains to be at the IDS-cement-restoration complex. In fact, the IDS layer was conditioned using tribochemical silica-coating and silanization in order to increase the adhesion between prepolymerized IDS and the resin cement. Apparently, this interface suffered form aging during thermo-mechanical loading. In this study, after removal of temporary restorations, IDS layer was not re-created as this procedure could affect the precise fit of the inlay. Surface conditioning methods with silane coupling agents other than 3-methacryloxypropyl trimethoxysilane coupling agent,  $\gamma$ -MPS, could increase the adhesion that needs to be further elaborated [45].

In earlier studies on veneers, the weakest link in adhesion seemed to be between the adhesive layer and the dentin [46-48]. In this study however with posterior inlays, adhesion to dentin was not impaired in the majority of the cases. This could be due to axial loading only whereas in laminates both shear and tensile forces are exposed to

the bonded interfaces. Nevertheless, also based on the high incidence of repairable failures, higher survival of the tooth itself could be expected with both materials tested for inlay restorations in molars.

## **5. Conclusions**

From this study, the following could be concluded:

1. The application of immediate dentin sealing significantly improved the fracture resistance of lithium disilicate inlays bonded to dentin.
2. Occlusal wear was more common with the multiphase resin composite inlays than with lithium disilicate after thermo-mechanical aging.
3. Multiphase resin composite inlays showed more irreparable failures and immediate dentin sealing did not improve its fracture resistance.

## **Acknowledgements**

The authors acknowledge Mr. P. Oosterwijk, B. van der Wal and A. van Elk of Dental Laboratory Oosterwijk/Elysee, Groningen, The Netherlands, for fabricating the ceramic inlays, and extend their gratitude to Ivoclar Vivadent, Schaan, Liechtenstein, 3M ESPE, St. Paul, USA, and Kerr, Orange, CA, USA for generous provision of some of the materials used in this study.

## **Conflict of interest**

The authors did not have any commercial interest in any of the materials used in this study.



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## Captions to tables and figures:

### Tables:

**Table 1.** The brands, types, chemical compositions, manufacturers and batch numbers of the main materials used in this study.

**Table 2.** Fracture strength results (Mean  $\pm$  standard deviation) (Newton) of experimental groups after thermo-mechanical aging and axial loading, minimum, maximum and Confidence Intervals (95%). Same lower-case letters in each column indicate no significant differences within each column ( $p>0.05$ ). For group descriptions see Fig. 1.

**Table 3.** Frequencies of failure modes after fracture test. Score 1: Fracture of the inlay; Score 2: Fracture of the inlay and enamel; Score 3: Fracture of the inlay, enamel and dentin, Score 4: Root fracture.

### Figures:

**Fig. 1.** Flow-chart showing experimental sequence and allocation of groups.

**Fig. 2** Probability plot with Weibull curves (95% CI) using maximum likelihood estimation, scale and shape values for all groups. 1: L-IDS<sup>-</sup>, 2: L-IDS<sup>+</sup>, 3: MR-IDS<sup>-</sup>, 4: MR-IDS<sup>+</sup>.

**Figs. 3a-b.** SEM images of inlays after thermo-mechanical aging from occlusal surfaces **a)** Lithium disilicate ceramic. Note the air-bubbles (\*\*) after the wear of the glaze layer (\*), **b)** Multiphase resin composite. Note the extensive wear (+) with small chippings on the occlusal surface (\*\*).

**Figs. 4a-b.** SEM images of a representative specimen from **a)** group L-IDS<sup>+</sup>. Note the fractured inlay (\*) with the IDS (Immediate Dentin Sealing) layer (\*\*) on the dentin surface (\*\*\*), **b)** group MR-IDS<sup>+</sup>. Note the interface between MR (+) and IDS (\*\*).

## Tables:

Brand	Type	Manufacturer	Composition	Batch number
<b>Clearfil SE Bond: Primer</b>	Primer	Kuraray Co., Tokyo, Japan	10-Methacryloyloxydecyl dihydrogenphosphate (MDP), 2-Hydroxyethyl methacrylate (HEMA), Hydrophilic dimethacrylate, dl-Camphorquinone, N, N-di-ethanol-p-toluidine, water	200022
<b>Clearfil SE Bond: Bond</b>	Bonding	Kuraray Co.	10-Methacryloyloxydecyl dihydrogenphosphate (MDP), Bisphenol A diglycidylmethacrylate (bis-GMA), 2-Hydroxyethyl methacrylate (HEMA), Hydrophobic dimethylacrylate, dl-Camphorquinone, N, N-di-ethanol-p-toluidine, Silanised colloidal silica	2T0038
<b>Tetric Evoflow</b>	Flowable composite	Ivoclar Vivadent, Schaan, Liechtenstein	Dimethacrylates (38% wt), barium glass, ytterbium trifluoride, highly dispersed silicon dioxide, mixed oxide and copolymer (62% wt). Additives, catalysts, stabilizers and pigments (<1% wt). Particle size: 40 nm (0.04 µm) - 3000 nm (3 µm). Mean particle size: 550 nm (0.55 µm)	S14454
<b>Glycerin Gel</b>	Glycerin gel	Johnson & Johnson, Sezanne, France	Glycerin gel	3099VA
<b>Durelon</b>	Carboxylate cement	3M ESPE, St. Paul, Minnesota, USA	Powder: Zinc oxide, stannous fluoride, tin dioxide. Liquid: Water and polyacrylic acid	525252
<b>CoJet Sand</b>	Particle for air-abrasion	3M ESPE	Aluminium trioxide particles coated with silica, particle size: 30 µm	446317 446317
<b>ESPE-Sil</b>	Silane	3M ESPE	Ethyl alcohol, methacryloxypropyl, trimethoxysilane	551520 550016
<b>Total-Etch</b>	Etching gel, 37% Phosphoric acid	Ivoclar Vivadent	37% phosphoric acid (H <sub>3</sub> PO <sub>4</sub> )	T20546
<b>Adhesive Universal</b>	Bonding	Ivoclar Vivadent	Methacrylates, ethanol, water, highly dispersed silicon dioxide, initiators and stabilizers	T28040 T24701
<b>IPS Ceramic Etching Gel</b>	< 5% Hydrofluoric Acid	Ivoclar Vivadent	<5% Hydrofluoric acid	T19032
<b>IPS Ceramic Neutralizing Powder</b>	Neutralizing powder	Ivoclar Vivadent	25-50% sodium carbonate, 25-50% calcium carbonate	T11686
<b>Monobond Plus</b>	Silane	Ivoclar Vivadent	Ethanol, 3-trimethoxysilylpropylmethacrylate, methacrylated phosphoric acid ester	T07775 T21454
<b>Variolink Esthetic</b>	Dual cure resin composite cement	Ivoclar Vivadent	Monomers: Urethane dimethacrylate, methacrylate. Fillers: ytterbium trifluoride and pheroid mixed oxide initiators, stabilizers and pigments. Particle size: 0.04-0.2 µm. Mean particle size: 0.1 µm. Total volume of inorganic fillers: approx. 38%.	T15625 T30447
<b>Lava Ultimate</b>	Multiphase resin composite (Shade A2)	3M ESPE	80% nano ceramic components with 20% of polymer matrix	498875
<b>IPS e.max Press</b>	Lithiumdisilicate (Shade A2)	Ivoclar Vivadent	SiO <sub>2</sub> , Li <sub>2</sub> O, K <sub>2</sub> O, MgO, ZnO, Al <sub>2</sub> O <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> and other oxides	R59340, R64197, R61630, R70382

**Table 1.** The brands, types, chemical compositions, manufacturers and batch numbers of the main materials used in this study.

Experimental Groups	n	Mean (SD)	Minimum	Maximum	Confidence Interval	
					Lower Bound	Upper Bound
L-IDS <sup>-</sup>	10	1358±506 <sup>a</sup>	861	2362	2068.2	2788.1
L-IDS <sup>+</sup>	10	2035±403 <sup>b</sup>	1499	2799	2301.5	3048
MR-IDS <sup>-</sup>	10	1861±423 <sup>c</sup>	1238	2746	1199.6	1798.3
MR-IDS <sup>+</sup>	10	1702±596 <sup>c</sup>	891	2644	993.6	1241.6

**Table 2.** Fracture strength results (Mean ± standard deviation) (Newton) of experimental groups after thermo-mechanical aging and axial loading, minimum, maximum and Confidence Intervals (95%). Same lower-case letters in each column indicate no significant differences within each column (p>0.05). For group descriptions see Fig. 1.

	Failure types				IDS present on tooth		
	Score 1	Score 2	Score 3	Score 4	Yes	No	N
L-IDS <sup>-</sup>		8			4	4	8
L-IDS <sup>+</sup>	1	3	3	3	10	0	10
MR-IDS <sup>-</sup>	2	2	5	1	4	6	10
MR-IDS <sup>+</sup>	4	2	2	2	10	0	10

**Table 3.** Frequencies of failure modes after fracture test. Score 1: Fracture of the inlay; Score 2: Fracture of the inlay and enamel; Score 3: Fracture of the inlay, enamel and dentin, Score 4: Root fracture.



Figures:

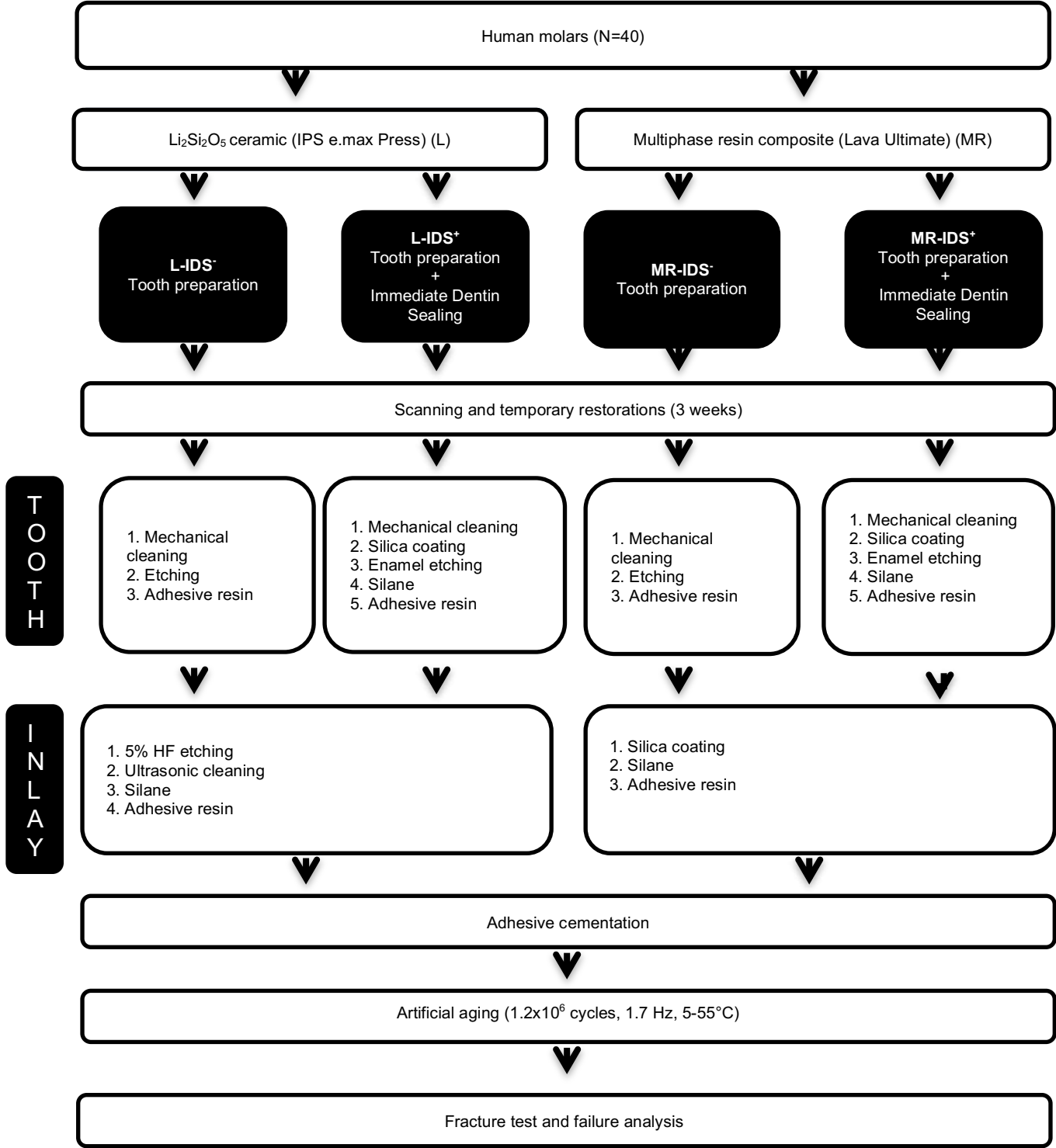
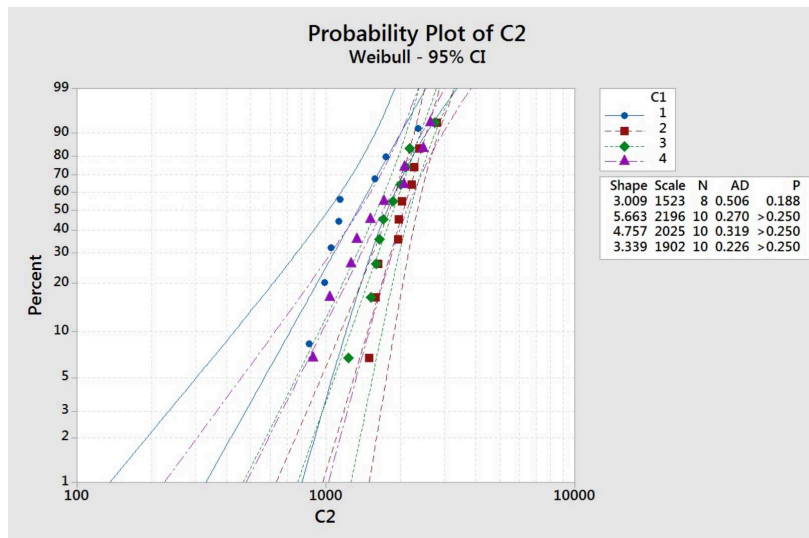
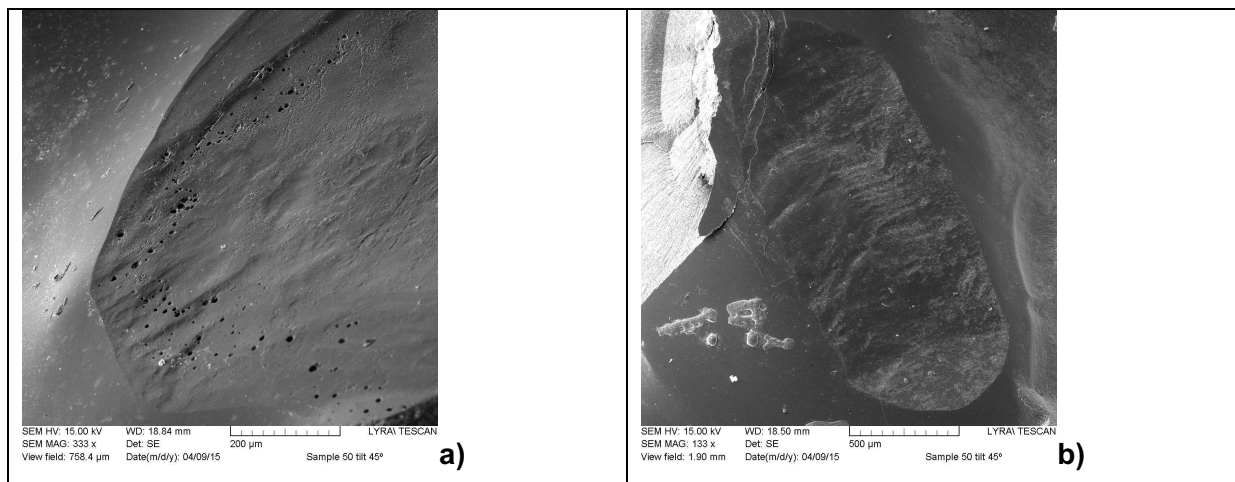


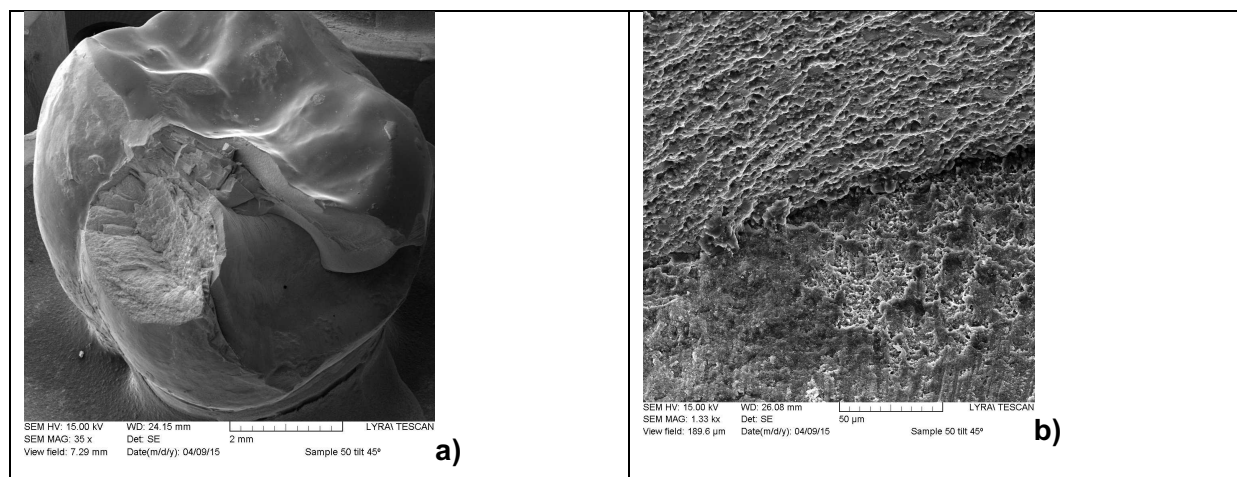
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